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FIRST QUARTERLY PROGRESS REPORT  
OCTOBER 1, 1980 - DECEMBER 31, 1980  
CPFF CONTRACT NO. 955733

DEVELOPMENT OF ADVANCED CZOCHRALSKI  
GROWTH PROCESS TO PRODUCE LOW COST  
150 KG SILICON INGOTS FROM A SINGLE CRUCIBLE  
FOR TECHNOLOGY READINESS

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## CONTENTS

	<u>PAGE</u>
I INTRODUCTION	1
II SUMMARY	2
III PROGRESS	2
A. CONSTRUCTION AND TEST	2
B. PROCESS DEVELOPMENT	3
C. CONTROLS AND AUTOMATION	3
D. ANALYTICAL STUDY	6
E. DOCUMENTATION	7
IV PROGRAM PLAN	7
V COST AND DIRECT LABOR DATA	8

## FIGURES

FIGURE 1 CONSTRUCTION AND TEST PHASE SCHEDULE	9
FIGURE 2 PROGRAM PLAN	10
FIGURE 3 TOTAL INCURRED COSTS	11
FIGURE 4 DIRECT LABOR HOURS	12

## **I INTRODUCTION**

The program for "Advanced Czochralski Growth Process to Produce Low-Cost 150 kg Silicon Ingots from a Single Crucible for Technology Readiness", can be divided into five general task categories:

- A. Construction and Test - to provide a modified CG2000 grower for process development and sensor/automated controls development.
- B. Process Development - for accelerated growth, accelerated recharge and yield/cost improvement.
- C. Controls and Automation - for sensor development and microprocessor controls integration to the Mod CG2000.
- D. Analytical Study - for purity analyses and solar cell fabrication.
- E. Documentation - for reporting, economic analysis, and process specification.

All of these tasks overlap in time phasing (see program plan). However, the construction and test of the modified CG2000 is required in the early portion of the program.

The contract goals require a crystal grower capable of pulling five crystals, each of approximately 30 kg in weight, 150 mm diameter, from a single crucible with periodic melt replenishment. The necessary grower modifications and process development tasks include:

- A. Overall equipment design, construction, and test
- B. Interface for automation with microprocessor controls
- C. Recharge rate of greater than 25 kg per hour with chunk or granular silicon using a recharge hopper
- D. Sensor development access ports in the chambers for melt level, melt temperature, and crystal diameter
- E. Radiation shield to accelerate growth
- F. Modified growth chamber suitable for use as a production facility
- G. Throughput capability of 2.5 kg per hour of machine operation using a radiation shield.

## II SUMMARY

Design, release for manufacture, and procurement of a modified CG2000 RC crystal grower proceeded during this quarter. The construction, installation, and test of this machine is expected by March 1, 1981.

The process development work will begin when the grower is completed. All of the hot zones, polycrystalline silicon, crucibles, dopant, seeds, and other supplies were quoted and purchase orders issued. Several exhaust gas analysis system equipment specifications and quotations were received and are under study.

Process control requirements have been defined and design work on the melt temperature, melt level and continuous diameter control begun. Sensor development included assembly and testing of a bench prototype of a diameter scanner system.

The overall program is proceeding on schedule.

## III PROGRESS

### A. Construction and Test

As part of the Technology Readiness goals, unilateral modifications to JPL Contract No. 954888 were issued to design a modified Hamco CG2000 RC crystal grower starting in late June, 1980.

By the October - December time frame, the design program was peaking and scheduled for completion in December, 1980. This contract, No. 955733, provided for the construction and testing of the Mod CG2000 RC by March, 1981. The design phase program of 954888 and the construction/test phase program of 955733 were constructed to dove-tail and minimize lead times for machine construction.

By November, the hot zone designs were completed, quotations received, and purchase orders placed for delivery in late January, 1981.

Similarly, the pull chamber system, furnace tanks, baseplate and frame, power supply, and crucible lift mechanism were all released for purchase and/or manufacture.

The baseplate, frame, crucible lift mechanism and power supply were completed by, or delivered to, Hamco in December, 1980.

The designs for the ancillary mechanical and electrical modifications were completed by mid-December and released for expected completion in January/February, 1981.

During this reporting period, the design was completed, and all quotations, purchase orders, and documentation for construction released to the Hamco manufacturing department.

The modified grower prototype is expected to be built and tested by late February, 1981.

#### **B. Process Development**

Orders were placed for the supply of both 15" and 16" diameter graphite hot zones and insulation packages. Initial process development will be undertaken using 15" diameter piece parts and will be followed by process development using 16" piece parts.

A supply of polycrystalline silicon feedstock was ordered from Dynamit Nobel of America at a cost of \$82.50 per kilogram. Approval for the transfer of 48 kg of polysilicon from the concluded JPL contract number 955270 to this contract was received from JPL. This transfer helped to offset the increase of \$2.50 per kilogram over the cost estimate calculated into the original contract proposal.

Quotations were received for the supply of 15" and 16" diameter opaque quartz crucibles at a cost of \$325 and \$391 respectively. An order was placed for five 15" diameter crucibles with Quartz Scientific. A supply of boron dopant was also ordered from Hemlock Semiconductor.

#### **C. Controls and Automation**

The Mod CG2000 grower being constructed under this contract will be equipped with a Kayex-Hamco Automatic Grower Logic (AGL) computer-based control system.\* Tasks within this contract address the development of sensors for integration with the AGL control system that will enhance its control capability for large diameter, high volume production of solar cell material. Sensors being developed

\*Automatic Grower Logic is a proprietary development of the Kayex Corporation.

include melt level, melt temperature and ingot diameter.

The following goals have been established for the control and automation tasks:

- Automatic setting of dip temperature based on direct measurement of melt temperature;
- Growth of neck, crown, body and final taper with a minimum of operator judgement and input;
- Maintenance of ingot body diameter to a tolerance of  $\pm 0.5$  mm including the effects of both melt level and diameter control.

## 1. Automation

As mentioned above, the automatic control function will be performed by the Kayex AGL system. System operation includes the following functions:

- a. Meltdown - Timed recipe in heater power level. Operator may initiate a final-time extension if melt is not complete.
- b. Stabilization - Closed loop control of melt temperature to stabilize at proper temperature for seed dip.
- c. Neck and Crown Growth - Programmed growth of neck and crown of predetermined and reproducible shape.
- d. Body Growth - Closed loop control of crystal body diameter and control of heater power level to maintain constant average seed lift rate.
- e. Final Taper - Initiated automatically after a predetermined length of ingot has been grown. Or taper may be initiated at an earlier stage by operator input.

## 2. Sensor Development - Melt Temperature

Accurate determination of the melt temperature prior to seed dip is judged to be necessary in order to insure that the initial conditions for crystal growth are reproduced. Optical pyrometry is the traditional approach for noncontact measurement of temperatures in the range of the silicon melting point. Single-color or total-intensity pyrometers are sensitive to clouding of the furnace viewing window or silicon monoxide "smoke" above the melt surface in the crucible. Two-

color pyrometers, which determine temperature from the ratio of the intensity of radiation at two different wavelengths, are less susceptible to error from these sources.

The approach being taken is to evaluate a commercially available two-color pyrometer for accuracy and reproducibility. Work to date has included the design of mountings and viewports that will allow viewing of the melt at near normal incidence. This geometry will minimize the amount of reflected radiation seen by the pyrometer and result in significant reduction of this error source.

### 3. Sensor Development - Melt Level

The traditional control approach uses a fixed ratio applied to the seed lift rate to control the crucible lift rate. Inaccuracies in crucible geometry or crystal diameter result in inaccuracies in the ratio and variations in the melt level from the nominal.

Three alternative approaches to direct melt level control will be evaluated.

- a. The first approach utilizes the output of a fixed laser reflected off the silicon surface and then detected by a position sensitive photodetector. Changes in the position of the melt level result in changes in the position where the reflected laser light strikes the detector. The detector output is suitably processed to yield a position signal which will be used as an error input to the crucible lift motor controller.
- b. The second approach derives melt level from a scanner system which is used for both melt level and diameter measurements. This approach is a proprietary Kayex development.
- c. The third approach uses a crystal weight output to drive the crucible lift and maintain the desired melt level.

### 4. Sensor Development - Ingot Diameter

Continuous measurement of the ingot diameter from neck through body growth will allow implementation of closed loop diameter control through these critical stages of crystal growth. The proprietary scanner system for determination of

crystal diameter and melt level is being implemented.

During this reporting period, construction of equipment for bench testing of the diameter scanner was completed. Alignment and test of both the optical and electronic components is underway.

Control requirements (goals) for the process have been defined and the design work for the integration to the Mod CG2000 of a Kayex-Hamco AGL computer-based control system is proceeding.

#### **D. Analytical Study**

The analytical task consists of three areas:

1. Purity Analyses of Silicon - The control of silicon purity will be monitored by chemical impurity analysis of selected samples from feedstock, grown ingots, and residual melt.
2. Solar Cell Fabrication and Analysis - Selected ingot material from all 150 kg runs will be sliced into wafers and, along with several control samples, fabricated into solar cells. These cells will also be tested for solar efficiency.
3. Furnace Atmosphere Analysis - A gas chromatograph and sampling system will be used to monitor the oxygen, and other possible impurities.

The activity in this area during this reporting period was:

1. For the 15" and 16" opaque quartz crucible from Quartz Scientific, arrangements were made for samples of quartz. Purity analysis will be made of the sample. The data obtained will be used as the comparator when impurity build-up analysis is made on each of the used crucibles. Quartz Scientific will also provide an analysis of impurity in the sand used to fabricate the crucibles.
2. Evaluation of three estimates received for the supply of a suitable gas chromatography analysis system incorporating a suitable gas sampling method are ongoing. Gas chromatography analysis techniques have the capability of

analyzing and differentiating between the quantities of carbon monoxide and nitrogen present in the crystal grower exhaust gas. Continuous monitoring of a specific gas impurity would not be possible and each total analysis cycle will take approximately 15 minutes, i.e. four total gas analysis cycles per hour.

The three G.C. systems quoted are as follows:

Hewlett Packard quoted a HP5712FL system at \$13,860 and a HP5840A system at \$17,880. Hewlett Packard declined to quote a system capable of sampling the crystal grower off gas into either of their systems.

Perkin Elmer indicated an approximate cost of a Sigma B system for \$15,000 and a Sigma 1B system for \$17,000. They located a suitable sampling system manufactured by Valco Instruments Co. of Houston. The cost would be approximately \$400. Perkin Elmer have been informed that a budget cost of \$10,000 was calculated into the contract RFP and, as such, could not be increased. They are currently evaluating the possibility of supplying a total system at this budget cost.

Baseline Industries, Inc., Lyons, Colorado quoted \$9,320 excluding installation and training costs for 1030 series gas chromatograph incorporating a Hewlett Packard 3390A Integrator. Installation and training would probably cost approximately \$1,000.

It is felt that either a Perkin Elmer or a Baseline Industries gas chromatograph appears to represent the most practical and cost effective system for our application. Both systems include a gas chromatograph reporting integrator and gas sampling system.

#### E. Documentation

Preliminary economic analyses show a \$20/kg CZ add-on cost from a single 16" crucible, 15 cm diameter crystal, 5 crystals x 30 kg each, and 2.5 kg/hr throughput. Detailed calculations will be presented in the February report and the next Project Integration Meeting.

#### **IV PROGRAM PLAN**

The overall program proceeded well and is on schedule. The construction and test

phase schedule update is included in Figure 1. An updated program plan is included in Figure 2.

#### V COST AND DIRECT LABOR DATA

The total incurred costs and direct labor graphs are updated and included in Figures 3 and 4.

	<u>Previous Total</u>	<u>Current Month</u>	<u>Total To Date</u>
Costs	\$ 22,417	\$ 15,874	\$ 38,291
Man-Hours	414	276	690

PROGRAM PLAN JPL CONTRACT #955733

BUILD AND TEST PHASE FOR MOD CG2000-RC  
FOR TECH. READINESS JPL #955733

WEEK ENDING

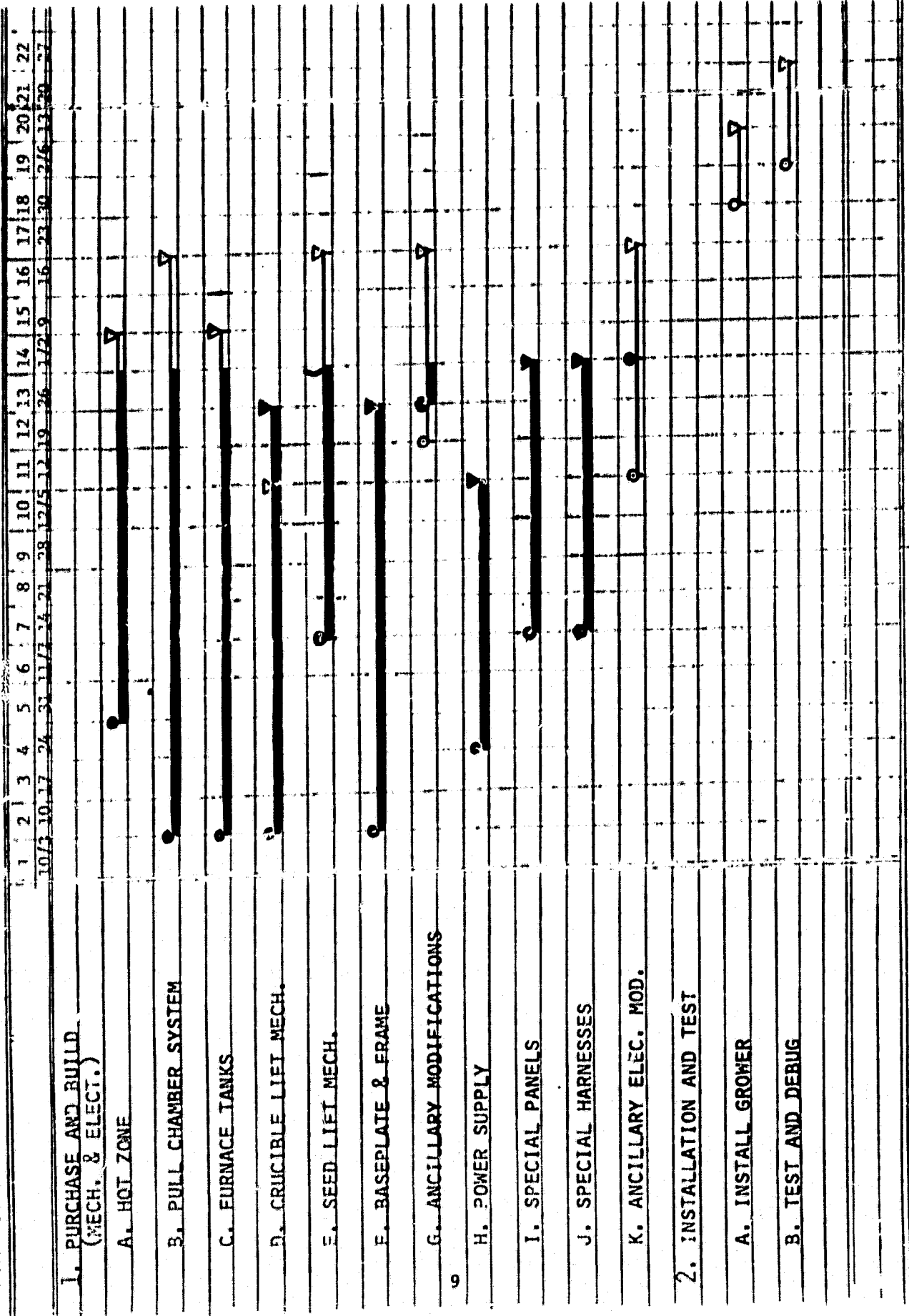


Figure 1

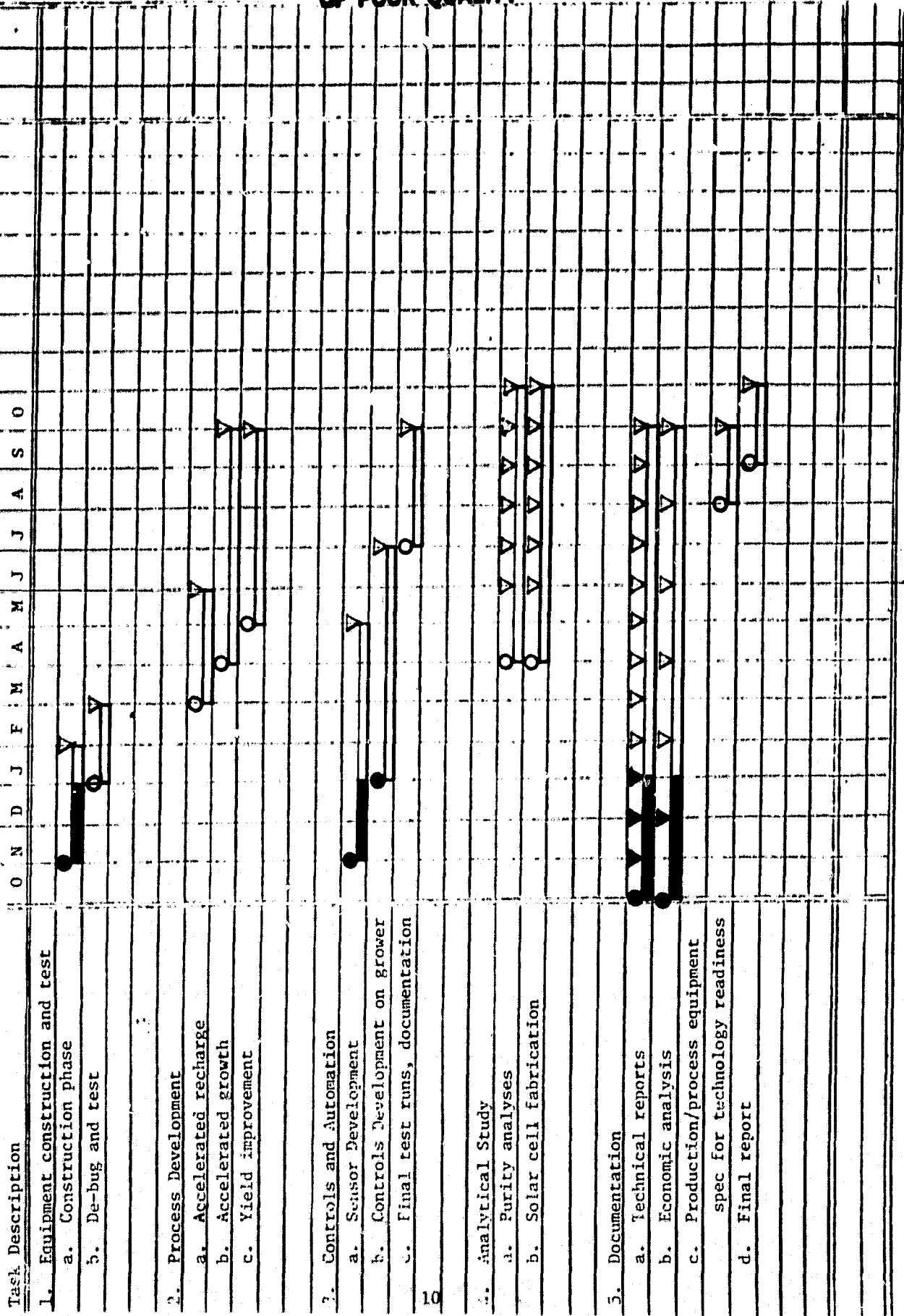
Project Title:  
Advanced Czochralski Growth  
for Technology Readiness

Program Plan, Revision No. 1

JPL CONTRACT #955733

Kayex Corporation  
January 20, 1981

Months Elapsed



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Figure 2

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ADVANCED C2 GROWTH  
TECHNOLOGY READINESS

KAYEX CORPORATION  
DOE/JPL 955733

TOTAL INCURRED COSTS

—x— PROTECTED  
—o— ACTUAL

TOTAL CONTRACT COSTS x \$1000

MONTHS ELAPSED

← 1980 → ← 1981 →

FIGURE 3

No 19-0210 (R 2470-10) 10 Squares to the inch - Made in U.S.A.

VERNON McMILLAN, INC. ELIZABETH N.J. 07208

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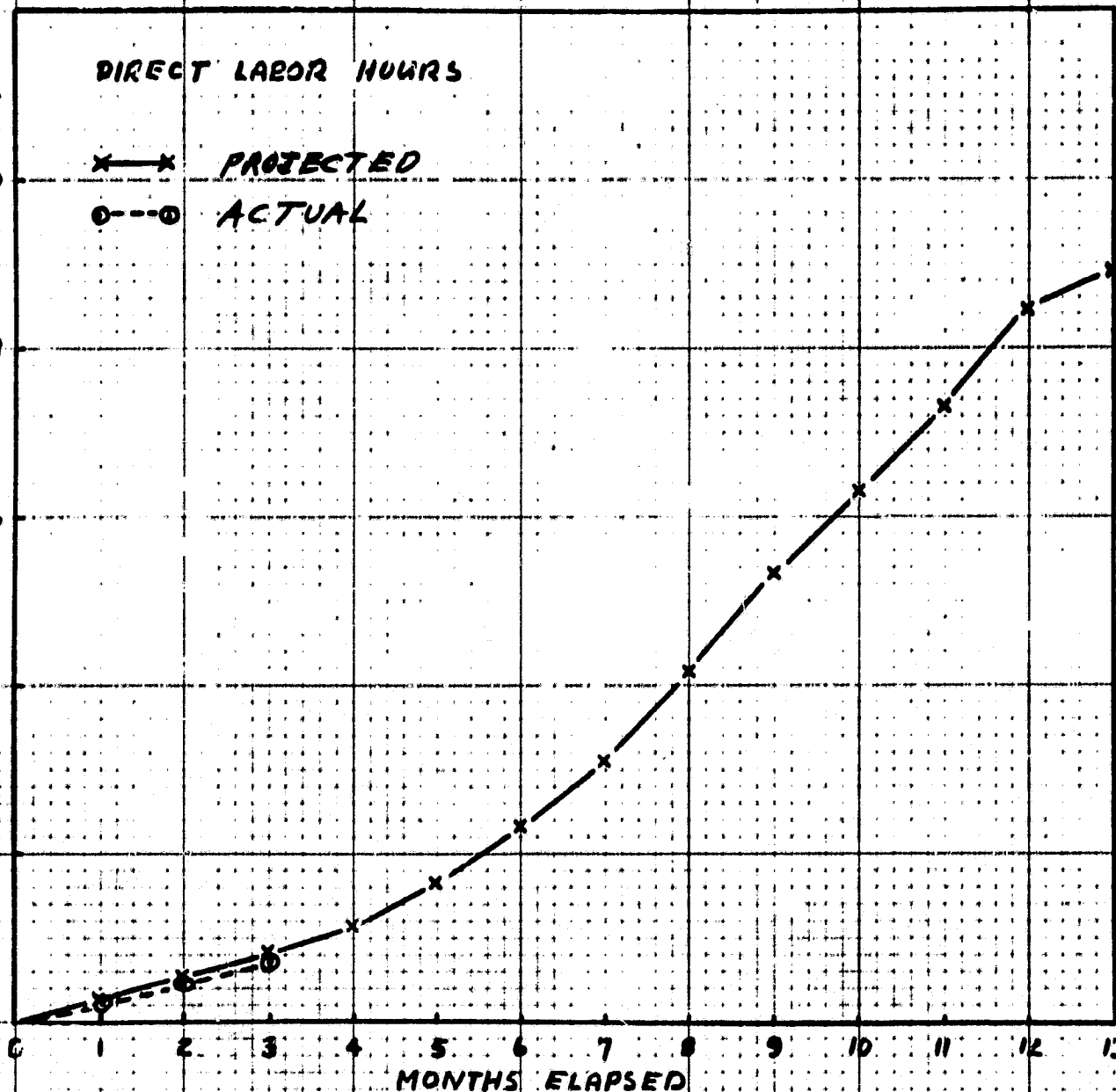
ADVANCED C2 GROWTH  
TECHNOLOGY READINESS

KAYEX CORPORATION  
DOE/JPL 955733

DIRECT LABOR HOURS

x-x PROTECTED  
o---o ACTUAL

DIRECT LABOR (1000 HOURS)



← 1980 → ← 1981 →

FIGURE 4